

CHARACTERIZATION OF AMORPHOUS SILICA AND CRYSTALLINE
SILICA FROM RICE HUSK ASH ON WATER FILTRATION APPLICATION

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DEDICATION

*“For my beloved mother RAHIMAH BINTI ABU BAKAR,
my lovely father ZAINAL BIN SAMSUDIN,
my dear brother MUHAMMAD SHAFIQ BIN ZAINAL
and my sweet sister NUR SHAFINAZ BINTI ZAINAL.
With every prayer and endless support.*

For my lovely friends who always encourage and be with me in every state”



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ABSTRACT

The presence of water pollution which consists of heavy metals, fecal and others has produced a major problem. These can lead the water to the toxicity and the impurity of water will be disrupted. Therefore, it would not be safe to drinks and could be threatening to live health. In addition, the current market now is dealing with high-cost production to develop ceramic membranes and has been using expensive material to make the filtration system works. In order to challenge the issue, the preparation of ceramic water filtration at low-cost production and using an effectively silica from natural waste rice husk was evaluated. Rice husk was fired at 700°C and 1000°C respectively and produced rice husk ash which mutated to amorphous and crystalline silica. Five samples were fabricated after been mixed with the compositions of rice husk ash, kaolin clay, and wheat flour, used at 40:40:20 ratios by weight respectively. The fabrications of the ceramic membrane were conducted by using dry pressing. The samples then were dried in the oven at 60°C for 1 hour followed by sintering at 1000°C respectively. These samples (OO, C1, C2, N1, and N2) were tagged based on unwashed and washed material with the chemical. The properties of silica which are the microstructure and pore size, from rice husk ash were obtained by using Scanning Electron Microscopy (SEM), X-Ray Fluorescence (XRF) and x-ray diffractometer (XRD). Effect of silica content in ceramic filtration membrane was investigated and characterized in term of porosity, density, water absorption, membrane hardness, pore size, flow rate, the turbidity of water (before and after filtration) and pH value. From the result, sample C2 was the best option to support the objective by 98.60% silica content, 64.82% of porosity, 1.1433 mg/cm³ of density, 40.59% of water absorption, 171.0 Hv of hardness, 0.177 l/hr of flowrate and pH of 7.62 of water after filtration. In general, the quality of the ceramic filter membrane is reliant on the raw material, while the flow rate and water clarity are dependent on the pore size of the filter membrane.

ABSTRAK

Kehadiran pencemaran air yang terdiri daripada logam berat, najis dan sebagainya telah menghasilkan masalah besar. Ini boleh menjadikan air lebih bertoksik dan kejernihan air akan terganggu. Oleh itu, ia boleh mengancam kesihatan dan mengganggu kualiti air. Di samping itu, pasaran semasa kini telah berhadapan dengan kos pengeluaran yang tinggi untuk membina fabrikasi membran seramik dan telah menggunakan bahan yang mahal untuk membuat sistem penapisan. Untuk mengatasi isu ini, penyediaan penapisan air seramik pada kos pengeluaran yang rendah dan menggunakan silika dari sekam beras buangan asli dikaji. Sekam beras dibakar pada suhu 700°C dan 1000°C dan menghasilkan abu sekam padi yang bermutasi kepada silika amorfus dan kristal. Lima sampel telah dibuat selepas dicampur dengan komposisi abu sekam padi, tanah liat kaolin, dan tepung gandum, dengan menggunakan nisbah mengikut berat masing-masing 40:40:20. Fabrikasi membran seramik dilakukan dengan menggunakan tekanan kering. Sampel kemudiannya dikeringkan di dalam ketuhar pada 60 °C selama 1 jam diikuti oleh pensinteran pada suhu 1000 °C. Lima sampel iaitu (OO,C1,C2,N1 dan N2) ditandakan berdasarkan bahan yang tidak dibasuh dan dibasuh dengan bahan kimia. Sifat silika dari abu sekam padi diperoleh dengan menggunakan mikroskop elektron pengimbasan (SEM), X-Ray Fluorescence (XRF) dan x-ray difraksiometer (XRD). Kesan kandungan silika dalam membran penapisan seramik diselidik dan dicirikan dari segi keliangan, ketumpatan, penyerapan air, kekerasan membran, saiz liang, kadar aliran, kekeruhan air (sebelum dan selepas penapisan) dan nilai pH. Berdasarkan hasilnya, sampel C2 merupakan pilihan terbaik untuk menyokong objektif dengan mencapai 98.60% kandungan silika, 64.82% porositi, 1.1433 mg /cm³ ketumpatan, 40.59% penyerapan air, 171.0 Hv, 0.177 l/ jam dan pH 7.62. Secara umum kualiti membran penapis seramik bergantung kepada bahan mentah, manakala kadar aliran dan kejelasan air bergantung kepada saiz liang membran penapis.

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LIST OF SYMBOLS AND ABBREVIATIONS

°C	-	Degree Celsius
µm	-	Micrometre
Al ₂ O ₃	-	Aluminium oxide
ATS	-	Applicant Tracking System
C	-	Carbon
CaO	-	Calcium Oxide
Fe	-	Iron
K ₂ O	-	Potassium Oxide
l/hr		Litre per hour
Mg		Magnesium
mm		Millimetre
Na		Sodium
P ₂ O ₅		Phosphoric Oxide
RH	-	Rice husk
RHA		Rice husk ash
SEM		Scanning Electron Microscope
SiO ₂		Silica dioxide
Si ₃ N ₄		Nitride
UTHM		Universiti Tun Hussein Onn Malaysia
W _s		Submerge in liquid
W _d	-	Dry condition
W _w	-	After been immersed in liquid
XRD	-	X-Ray Diffraction
XRF	-	X-Ray Florescence

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CHAPTER 1

INTRODUCTION

1.1 Background of study

About the last decades, the exponential population and social civilization expansions, different in cycle of life and resources that have been used with extended advances of the industrial and technologies have to generate to the rapid growth of modernization and metropolitan (Shalaby *et al.*, 2017). The increase in industrial activities has greatly affected the changes in ecosystems which this has affected to the threat of environmental health of humans. Among the many cases of pollution, the contamination of water ranks the highest place.

Thus, to reduce the health and disease issues, water purification needs to be done by using the water filter. It is impossible to remove 100% of fecal, but there is a way of minimizing the contamination of water. There are a few possible ways which are a physical barrier, chemical process or a biological process by filtering the water to residue a cleanse for a resolution. For examples, providing agricultural drainage system, save in drinking water, protected from waterborne disease and the safe use of ponds and swimming pools.

One of the environmental sustainability targets is recycled back the agricultural waste products (Pandey *et al.*, 2012). The agricultural waste products will be used in water treatment and if well entrenched, it will be useful to those in the poor communities. Silica that present from the husked rice is used to enhance the production of existing filter since the worldwide annual rice husk output is about 80 million tones and over 97% of the husk is generated in the developing countries.

Malaysia is one of them. According to the statistics compiled by the Malaysian Ministry of Agriculture, 408,000 metric tonnes of rice husk is produced in Malaysia each year. (Noor Syuhadah & Rohasliney, 2012).

The rice industry produces substantial amounts of waste materials. Most of the waste materials originated in the rice production cycle which includes rice husk, rice straw, and rice husk ash. Rice husk is the external of the rice seed layer, removed during the grinding process. For every 4 tons of harvest rice, about 1 ton of rice husk is produced which is about 20% to 30% of the grain weight. Rice straw is the stem of the plant that sinistrial as the remainder in the field, after harvesting the grain (Lim J. *et al.*, 2012). For each kilo of paddy rice about 0.41-3.96 kg of rice straw are assembled (Li *et. al.*, 2017). Rice husk ash produced from the rice husk that has been fired.

To obtain high silica content, the combustion process according to the specification of amorphous content should be performed. After the combustion process, this material can produce 80% -95% of silica mineral. This material is also very easy to obtain and requires a low cost. The silica content found in rice husk is widely used in electronics, ceramics, plastic, rubber, and photoelectric industries. Silica from the rice husk has been used as a heat exchanger agent and composite additives (Zhang *et al.*, 2010).

Today in the market stream, although synthetic silica content has been used extensively, the use of silica from natural materials is still relevant due to its low cost and environmentally friendly. In this study, several tests have been conducted to investigate and study the structure and properties of the silica.

The application of silica can be found in the various field. This is because it is a safe chemical substance. Various applications have been used in various sectors. Among others are electronic, mechanical, art and even *zeolite* manufacturing. Silica products are commercialized in the form of *sol-gel* and amorphous silica fumes. Burning the rice husk at high temperatures will lead to the formation of silica with a crystal structure (Della, Kuhn & Hotza, 2002).

For many years, membrane technology especially with a porous structure, has been the most contributor for water treatment application (Baker, 2012). Among of all types of membrane, polymeric membranes are recognized for its ability, of high porosity and easy performance.

1.2 Problem statement

Malaysia produces quite a large amount of agricultural waste that has a great potential to be exploited for good. Fabrication of water filter from this natural waste resources could help to reduce water pollution in Malaysia and offer a healthier lives. The presence of several water filters made of ceramic materials in the market shows the potential of it to clean and strain the unwanted substances.

But, there are a few problems with the current water filters that available in the market. Water filter is used in almost every house and has benefited many users. However, water filters that available in the market are very expensive, while there are still many people who cannot afford to have a quality water filters especially for those who live in rural and urban areas. In fact, there are societies that still rely on water from rivers and wells. Moreover, the quality of water in state of odour, impurity and contaminant also are possible problems to look. Therefore, in this work, the water filter has to fill the requirements by improving the granular structure, chemical stability, and local availability at almost no cost to improve the quality of water.

Therefore, this research aims to study and investigate the potential of Silica (SiO_2) used in the water filter fabrication. The research methodology has been conducted in accordance with the established standards.

1.3 Significant of study

There are numerous technology of water treatment that can be obtained in the market. The expeditious of population's growth due to economic development has increased the demand for energy. Prior to these statements, the natural resource base and water scarcity have been threatened by social sector. By producing the ceramic water filtration membrane would be one of the best water filter due to its reliability and others as following factors :

- i. The sustainability factor
 - This ceramic filtration membrane water filter is made from dumped natural waste resources which are rice husk. Thus, it contributes to environmentally friendly by minimizing the rice husk waste.
- ii. The cost factor
 - The main material used for ceramic water filter in the current market is mostly using synthetic material. Therefore, by using ceramic water filter fabricated from rice husk ash, it is relatively cheap because of the maintenance cost and operation costs are lower compared to the other materials.
- iii. Performance factor
 - The layer filtration procedure is adaptable. It may be utilized in the partition, concentration, and purification of different materials over a wide scope of the industries and households.

1.4 Objectives of study

There are several objectives that have been identified as follows :

- i. To identify the potential of Silica (SiO_2) from the rice husk ash to be used in the water filter.
- ii. To investigate the effect of using amorphous and crystalline Silica (SiO_2) on flow rate, turbidity, and pH value of water after filtration.
- iii. To investigate the effect of using amorphous and crystalline Silica (SiO_2) on porosity, density, and hardness of the filtration membrane.

1.5 Scope of the study

Through this project, there are several tasks that have been done. The following are the scopes of the study that has been assigned to facilitate this study :

- i. Silica powder was produced from the rice husk through a thermal treatment at 700°C and 1000°C.
- ii. The rice husk ash was sieved to obtain the particle size of 63µm for rice husk.
- iii. The binder used were wheat flour and kaolin clay with 25 µm of particle size.
- iv. The ceramic water filter membrane was fabricated by using dry pressing technique.
- v. The specimen was sintered at 1000°C.
- vi. The performance and characteristic of the ceramic water filter were measured and analyzed by microstructural analysis, porosity and density test, flowrate test, hardness test, and turbidity test.



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CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter will discuss about the previous study which focus on the ideas and strategy used by the researcher that related to this research. Detail information about the ceramics, silica, rice husk and filter element and the water filter will be discussed.

2.2 Ceramic

Ceramic is derived from the Greek word *keramikos*, which means 'clay'. Ceramic is produced at a high temperature of 500°C. There are also some studies that states ceramics came from the word *keramos* which means any yield made of clay. However, both meanings are the same (Kingery, 1976).

Ceramic is a polycrystalline material and usually contains a mixture of the solid phase. The raw material is in the form of powder, formed and applied to heat treatment which is suitable to produce a strong and useful body. Crystal structures are complex structures compared to other materials. Ceramic contains several different elements of size. The bonds between atoms are covalent bonds and are stronger than metal bonding. Moreover, in terms of its hardness, the properties of heat in ceramic like high melting temperature, high hardness, poor conductivity, high moduli of elasticity, chemical resistance and low ductility are better than metal.

Many composites, such as glass fibers and carbon fiber, which contain ceramic materials, are not considered as part of the ceramic family.

The earliest ceramic products made by humans are pottery items such as plates, statues, pots, and others. These products can be produced without a mixture of other materials or with a mixture of other materials such as silica, hardness or sintered in high temperatures. Ceramics also can be glazed and fired for the production of smooth, coloured, porous surfaces by using amorphous ceramic coating on the top of the ceramic substrates (Degarmo *et al.*, 2012).

In line with modern ceramics, these materials are divided into oxide and non-oxide ceramics. Oxide ceramic consists of oxygen-containing substances like alumina (Al_2O_3), silica (SiO_2), zirconia (Zr_2O_2) and barium titanate (BaTiO_3). This type of ceramic material exists naturally in aid and minerals. Non-oxide ceramics include nitride (Si_3N_4 , TiN , BN) and carbide (SiC , TiC , B_4C). These materials are synthesized using natural raw materials or chemically. The next classification of modern ceramics is more to its functions and its field of applications (Kingery, 1976). Currently, ceramic materials are used for domestic, industrial, building, ceramic and the latest in engineering which is in semiconductor (Degarmo *et al.*, 2012).

In the ceramics industry, the raw materials used are clay minerals. Other substances such as fine sand, snail shell, flux minerals, bone, rice husk, coconut husk, and so on are mixed to avoid cracking and contraction problems during combustion. The addition of other materials can also improve the quality of the product. The ceramic products made from raw materials as mentioned are classified as traditional ceramics. Whereas the ceramic industry, focuses on ceramic production that provide special and sophisticated features and belong to the modern ceramics or advanced ceramics. It is also can be produced either from natural raw materials or synthetics where the fabrication process involves modern technology (Kingery, 1976). Figure 2.1 shows the water filter produced from ceramic based (Barbara Hepworth, 2013).



Figure 2.1 : Ceramic water filter (Barbara Hepworth, 2013)

2.2.1 Ceramic properties

Glass materials are classified as ceramic materials. It is fragile and cannot experience any plastic deformation, causing the material to be ineffective. In addition, the properties of this material whose pores, microscopic shafts and microscopic defects that act as a pressure proponent will cause tensile strength to decrease. Thus, it will give the ceramic material a tendency to failure as a whole and shatter over.

This material indicates a plastic deformity (elasticity not fracture). Such defective movements however occur slowly due to the rigid structure of the crystalline forming material and there are only a few slippery systems occurring. With non-crystalline material, the viscosity is the cause of plastic defects. Ceramic material is very strong under pressure and able to withstand at high temperatures. Its hardness also makes it suitable for corrosion and cutting edge for hardware (Jankowiak, 2016).

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